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Technical Note

Deep reactive ion etching of auxetic structures: present capabilities and challenges

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Abstract

Auxetic materials (or metamaterials) have negative Poisson ratios (NPR) and display the unexpected properties of lateral expansion when stretched, and equal and opposing densification when compressed. Such auxetic materials are being used more frequently in the development of novel products, especially in the fields of intelligent expandable actuators, shape-morphing structures, and minimally invasive implantable devices. Although several micromanufacturing technologies have already been applied to the development of auxetic materials and devices, additional precision is needed to take full advantage of their special mechanical properties. In this study, we present a very promising approach for the development of auxetic materials and devices based on the use of deep reactive ion etching (DRIE). The process stands out for its precision and its potential applications to mass production. To our knowledge, it represents the first time this technology has been applied to the manufacture of auxetic materials with nanometric details. We take into account the present capabilities and challenges linked to the use of DRIE in the development of auxetic materials and auxetic-based devices.

Keywords: auxetics, negative Poisson ratio, metamaterials, morphing structures

SQ1 (Some figures may appear in colour only in the online journal)

1. Introduction

When a material is stretched, there is normally an accompanying reduction in its width. A measure of this dimensional change can be defined by Poisson's ratio, $\nu = -d\varepsilon_{trans}/d\varepsilon_{axial}$, with ε_{trans} and ε_{axial} being the transverse and axial strains, respectively, when the material is stretched or compressed in
Q1 the axial direction. In a more general case, ν_{ij} is the Poisson ratio that corresponds to a contraction in direction 'j' when an extension is applied in direction 'i.' For most materials, this value is positive and reflects a need to conserve volume. Auxetic materials (or metamaterials) have negative Poisson ratios (NPR) and display the unexpected properties of lateral expansion when stretched, and equal and opposing densifi- Q2
cation when compressed (Lakes 1987, Evans *et al* 1991, He Q3
et al 2005, and Liu and Hu 2010). Natural (some minerals, Q4
skins, etc) and man-made (foams, Gore-Tex®, polymeric foams) auxetics have been described and very special attention has been paid, since their discovery, to searching for and developing auxetic structures designed and controlled on a molecular scale (Wojciechowski 1987, Evans *et al* 1991 and, more recently, Griffin *et al* 2005). Q5

It is necessary to note that auxetics, understood as materials and models of NPR, are not only geometries but also interactions with external conditions and constraints, such as negative pressure, proximity of certain phase transitions, specially woven materials, living tissues and their surroundings, and polydispersions, among other possibilities described in the seminal papers in this field of study (Wojciechowski 1989, Hirotsu 1991, Wojciechowski 2003, Nar- Q6 ojczyk and Wojciechowski 2010). In any case, auxetic materials and structures leading to auxetic behavior are being progressively employed in the development of novel products, especially in the fields of intelligent expandable actuators, shape-morphing structures, and minimally invasive implantable devices. Regarding smart actuators based on an auxetic structure, it is important to cite recent progress linked to the use of auxetic shape-memory alloys (SMA) in the Q7 development of deployable satellite antennas (Scarpa *et al* 2010), and research on the characterization of polyurethane foams with shape-memory behavior and auxetic properties, Q8 promoted thanks to several postprocessing stages (Bianchi *et al* 2010). In the area of medical devices, recent research has Q9 also assessed the behavior of a few auxetic structures for use Q10 in expandable stents (Tan *et al* 2011 and, more recently, Gatt Q11 *et al* 2014, and Mizzi *et al* 2014). Their application to other implantable biodevices is clearly a matter of current research.

Several auxetic materials and potentially auxetic structures, normally grouped under the terms 're-entrant' (Almg- Q12 ren 1985), 'chiral' (Prall and Lakes 1997), and 'rotating' (Grima and Evans 2000) in relation to the characteristics that Q13 promote their auxetic behavior, have been summarized in previous reviews and research. However, precise information regarding the values of Poisson ratios is not always provided, due to difficulties with simulating and manufacturing such complex geometries. Sometimes, just a scheme of their folding process, when submitted to uniaxial stresses, is provided, which proves to be of limited use for subsequent design activities. Recent comparative studies have tried to provide additional information on the relevant properties of different auxetic structures, in order to assist with material- and structure-selection tasks for the development of novel Q14 foldable-morphing actuators, structures, and devices (Álvarez Elipe and Díaz Lantada 2012).

Regarding the manufacture of auxetics, to our knowledge, the first successful attempt to obtain such auxetic structures in the microscale, for creating metamaterials with NPR, was made by soft lithography, leading to details and Q15 pores in the 100-micron range (Xu *et al* 1999). The lithographic process explained in this reference is interesting and promotes additional applications, such as microtubular structures.

However, for adequately exploiting the potential of auxetic metamaterials, an additional degree of precision is needed. The manufacture of polymeric sheets with auxetic nanostructures can prove useful for developing active selective membranes, whose pore size can be controlled in real time by applying uniaxial loads. Applications in the biomedical field (i.e. dialysis) and in energy (i.e. membranes for catalytic reactors) are worth exploring. By rolling such auxetic sheets, even easily implantable devices (i.e. stents) for minimally invasive surgical procedures can be obtained (Xu *et al* (1999)).

Tissue engineering, with interactions at a cellular and even molecular level, can also benefit from auxetic structures Q16 (Soman *et al* 2012), especially if these preliminary approaches are improved with more micro- or nanoauxetics, which allow us to obtain smaller clearances between the cells being cultured. During cell culture, uniaxial excitations of an auxetic scaffold lead to biaxial expansions and compressions of the tissue being grown, which promotes growth and can potentially control cell differentiation and tissue viability. However, using conventional photolithography or stereolithography for the manufacturing of two-dimensional (2D) auxetics, with typical distances between the lattices of the auxetic structure of more than 100 microns, leaves important clearances between cells and prevents them from interacting at the single-cellular level.

The special properties of auxetic materials can benefit both mechanical applications and auxetic devices for optoelectronics and telecommunications, because they require greater degrees of precision than is currently attainable in traditional micromachining. Some additional remarkable Q17 proposals for obtaining real mechanical metamaterials, with the finest details reaching hundreds of microns, include both subtractive approaches, such as UV laser ablation (Alderson Q18 *et al* 1999), and additive manufacturing procedures, such as stereolithography, digital light processing, and direct laser Q19 writing (Kadic *et al* 2012 and Bückmann *et al* 2012). Nor- Q20 mally 2D and 2D 1/2 auxetic structures are obtained by means of surface micromachining, chemical etching, laser ablation, and typical mass-production processes imported from electronics. Three-dimensional (3D) auxetics have more complex geometries and inner details, and require 3D additive manufacturing or 'layer-by-layer' processes, especially those support-less ones, such as selective laser sintering or selective Q21 laser melting.

In this study, we present a very promising approach for the development of auxetic metamaterials and devices based on the use of deep reactive ion etching (DRIE), which stands out for its precision and potential for use in mass production. To our knowledge, it represents the first time this technology has been applied to the manufacture of auxetic structures with nanometric details. In the following sections, we try to provide interesting details about the design and manufacturing processes we have used. We also discuss our main results and present capabilities, difficulties, and challenges regarding nanoauxetics.

2. Materials and methods

2.1. Design process

The re-entrant auxetic structure that we selected as an object of study was introduced by Almgren, and is the simplest (periodic) model used to describe auxetic foams (Almgren 1985). More recent and realistic models of foams can be found in other outstanding references (Pozniak *et al* 2013 and Q22 Chetcuti *et al* 2014), but for the purpose of our work we will Q23 use the aforementioned simple re-entrant structure as an

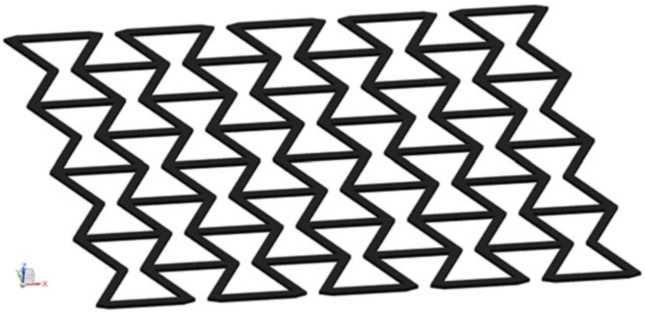

Figure 1. Two-dimensional (2D) auxetic structure used in our current research. Taken from the CAD auxetic library developed by Álvarez Elipe and Díaz Lantada (Prall and Lakes 1997), inspired by Almgren (Kadic *et al* 2012).

example. For our present research, we designed such a re-entrant auxetic structure using NX-8.5 (Siemens PLM Solutions), by first obtaining a unit cell and then using Boolean operations and 2D matrix replication. The planar auxetic was designed in the *XY* plane for subsequent extrusion along *z* direction, so the expected auxetic behavior should lead to both transversal contractions along the *y*-axis when compressed along the x direction, and expansions along the *y*-axis when tractions are applied along the x direction.

The process presented in this study may be suitable for the manufacture of several types of planar auxetics, many of which are collected in our recently developed library (Álvarez Elipe and Díaz Lantada 2012), but we have selected the re-entrant geometry shown in figure 1 because of its simplicity, well-understood behavior, and versatility. It is important to note that its Poisson ratio can be tuned by changing the relationships between the length, width, and re-entrant angle
Q24 of the unit cell (Lira *et al* 2009 and Sun and Pugno 2013). It
Q25 can be manufactured using both subtractive (typically 2D 1/2 micromachining) and additive approaches.

In addition, such geometry can be used as a basis for 3D auxetic structures, either by rolling, stacking, or using the
Q26 same principle but with 3D unit cells for the final develop-
Q27 ment of both planar and 3D auxetic-based devices (Yang *et al* 2011).

2.2. Manufacturing process

Plasma-phase or dry etching is a fundamental process in the semiconductor industry, commonly used for etching poly-silicon in surface micromachining and for forming hollow cavities in bulk micromachining. In short, plasma-phase etching involves the generation of chemically reactive neutrals and ions that are accelerated under the effect of an electromagnetic field towards a target substrate with protected and unprotected zones, thanks to the help of a physical mask. The reactive species are formed by the collision of molecules in a reactant gas with a cloud of energetic electrons excited by
Q28 an RF field. When the etching is purely chemical, powered by the spontaneous reaction of a neutral with silicon, the process is referred to as 'plasma etching.' The typical result of such an exclusively chemical etching is the so-called isotropic profile, which has a circular shape in its ideal case.

However, this kind of etching profile is usually not desired in common technological applications, which led to the development of different types of anisotropic etching techniques: If the ion bombardment of the silicon surface plays a relevant role in the promotion of the chemical etch reaction, the process is then called reactive-ion etching (RIE). The directionality of the accelerated ions gives RIE its characteristic anisotropy. Finally, a second plasma source above the substrate level can introduce a lot of additional power in the etching process onto the inductively coupled plasma (ICP) substrate. Q29

DRIE evolved from the need for an etching process capable of anisotropically etching high aspect ratio (AR) structures at mass-production rates. The process is similar to RIE, but the silicon substrate (a whole wafer or a smaller sample) is typically cooled to cryogenic temperature (−80 °C to −150 °C) for promoting condensation of reactant gases such as SiO_xF_y (see figure 2); this protects the sidewalls from both additional etching and redepositions, and helps obtain high ARs and almost-vertical and very smooth sidewalls. An alternative DRIE uses the 'Bosch process,' in which etching and deposition cycles alternate (Maluf 2000). During the last decade, DRIE technology development allowed the manufacturing of continuously smaller lateral dimensions deep into the nm range. Lateral dimensions of 100 nm–300 nm are common today (i.e. for photonic waveguides), and are often combined with structure heights from 200 nm up to 1 μm or more. At larger lateral dimensions, etching depths of up to 50 μm into silicon can be realized. A schematic overview of the process and the main etching chamber is shown in figure 2, and the real system used in this study can be seen in figure 3.

In spite of the detailed advantages, the cryogenic process for the anisotropic high-AR etching of silicon has some typical limitations, including the maximum AR attainable in channels and the possible length of on-fillets and cantilevers. As there is a certain thermal gradient, especially within very slim structures, the cooling effect, which comes from underneath the substrate, decreases in the upper areas of the structure while the etching in the ground runs deeper; in consequence, the sidewall protection in the upper area is limited. On the other hand, in very narrow channels or gaps, the number of vertically directed ions decreases exponentially with an increased etching depth; in consequence, the etch rate also decreases and, again, the etch process stops completely at a certain AR. Both effects limit the maximum AR attainable. A good value for the cryogenic DRIE process would be AR ~ 30. However, even higher values can be reached with some process modifications.

A crucial criterion for satisfying cryogenic etching results (smooth and vertical sidewalls, no residuals on any surface, right-angled edge in the ground, etc) is linked to the quality of the etching mask, or 'resist.' The ideal dry etching mask would have a very high selectivity (defined as the quotient of specific etch rates of substrate versus resist), very sharp and fine resolution at the edges, and good processability regarding

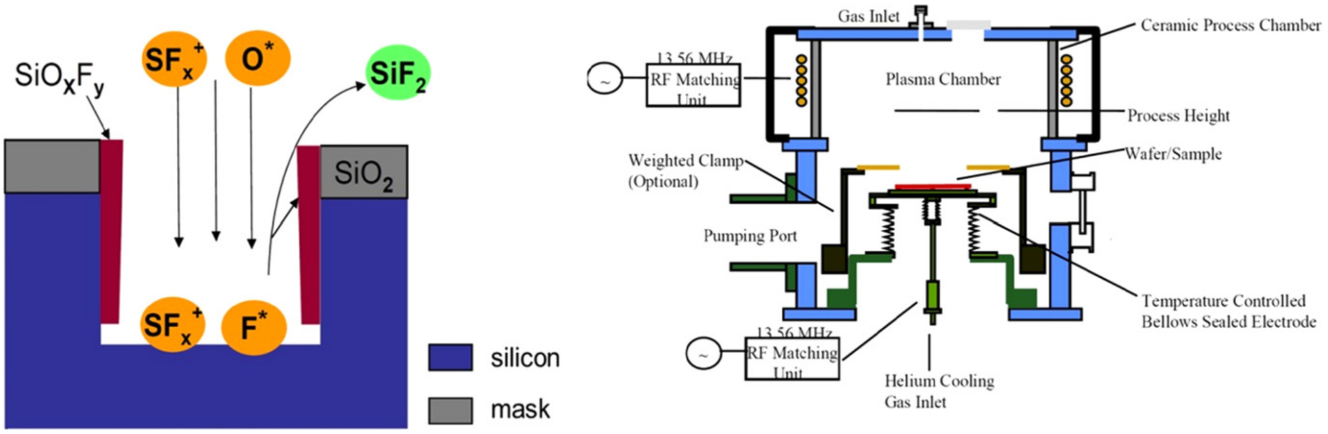

Figure 2. Process scheme and chemistry (left). Schematic view of the RIE/ICP process chamber (right).

Figure 3. Photograph of the DRIE facility at the Karlsruhe Institute of Technology.

the previous mask manufacturing process (electron-beam lithography). To satisfy these different properties, PMMA or
Q30 various AZ-photoresists can provide good compromises for the mask material. The typical process gas mixture, used in the cryo process, is SF_6 combined with O_2; fluorine ions or radicals are responsible for the main silicon etching process, whereas oxygen provides the required particles for the sidewall passivation. In addition to the previously described parameters of temperature, mask properties, and gas mixture, many other parameters determine and limit the etching results, including the RIE/ICP power value, the process pressure, the backside cooling pressure, the different gas flows, concentrations and ratios, and, last but not least, the substrate itself. All this makes the cryo DRIE process a highly complex and challenging technology, in reference to the stability, controllability, and reproducibility of established processes, as well as to the modification and technological development of new etching processes.

When using this cryo dry-etching process for silicon, the
Q31 auxetic structure is fabricated in the μm range, based on a SOI substrate with a silicon-device thickness of 1.0 μm and a 4.0 μm -thick box layer. The auxetic layout is written into a photoresist using laserlithography, and then etched into the device layer using the standard cryo process. Finally, the etched silicon structures are underetched, removing the oxide
layer with HF (wet etching). Q32

3. Results and discussion

An important complication of technologies that use multilayered wafers for selective etching is that large-area structures tend to deflect, due to stress gradients or as a consequence of the surface tension induced by trapped liquids, and attach to the substrate layer during the final rinsing process and drying step. It is likely that van der Waals forces and hydrogen bonding are responsible for such attachment and for keeping the microstructures firmly attached to the substrate. Once these stiction phenomena appear, the mechanical forces needed to release the parts from the substrates are usually large enough to damage the micromechanical structures (Gad-el-Hak 2003). Figure 4 shows examples of cantilever auxetics attached to the substrate after a preliminary DRIE aimed at process adjustment.

There are several interesting strategies that help avoid structure collapse and stiction to substrate. To reduce stresses and deformities, the auxetic structure can be manufactured in the form of a bridge instead of a cantilever, with two opposite sides linked to the unetched zone. Another possibility is using a thicker substrate to obtain 2D 1/2 auxetics with a much higher AR. Such an alternative would also promote a more adequate response to compressions and prevent the buckling phenomena detailed below. An additional option is reducing the device scale to obtain smaller cantilevers, taking advantage of the high precision attainable by DRIE.

A main challenge for future research is linked with the characterization of the actual auxetic behavior of the structures obtained, due to difficulties linked to manipulation and loading. The use of nanoindenters is common for the characterization of mechanical properties in nanometric lattice 3D
structures (Le Bourhis *et al* 2008). However, its application in Q33
characterizing the auxetics obtained is indeed challenging, as the auxetic should be placed vertically, and buckling would be a relevant issue when compressing such a 2D 1/2 structure.

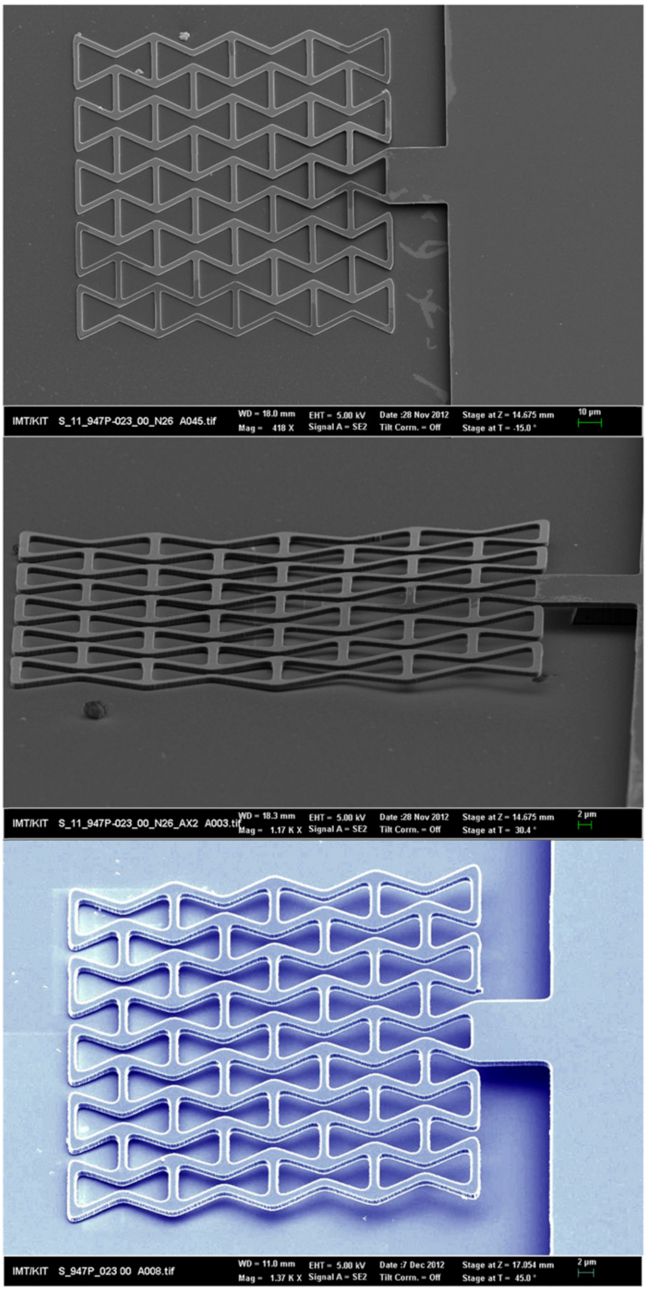

Figure 4. Different views of 2D auxetic cantilevers obtained by cryo DRIE.

Characterization by traction may be easier in this case, although common procedures based on atomic-force microcopy or traction- force microscopy, as typically used for cell
Q34 mechanotransduction (Wang *et al* 2007) and material prop-
Q35 erty assessment (Agero *et al* 2010) are also difficult to apply in this case. First of all it, would be difficult to reproduce the ideal boundary conditions for auxetic behavior characterization, such as traction in a desired direction that leaves the displacements in transversal directions free. In addition, it would not be possible to apply a desired deformation to a
Q36 complete edge of the auxetic structure, as the AFM tip would just apply the traction force to a unit cell of the structure.

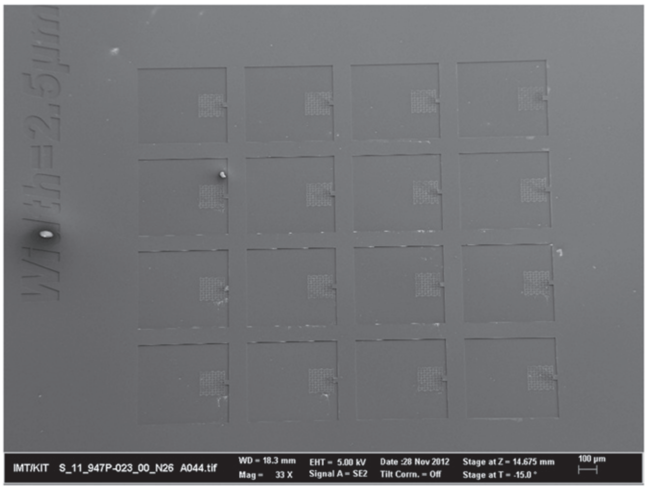

Figure 5. Several structures obtained in just one step: toward mass production of microauxetics.

Finally, as the whole structure is micrometric, the AFM tip would likely break during the process.

When considering the industrial expansion of auxetic-based microdevices and appliances, further research, and study tasks, DRIE provides interesting advantages compared to other prototyping techniques, because it allows mass production to be achieved once the process is adequately adjusted. As a preliminary example of this mass-manufacturing potential, figure 5 shows 16 copies of the auxetic structure produced in a single wafer, in a process that stands out for its accuracy and repeatability.

The structures obtained, which have an overall size of around $100 \times 100\,\mu\text{m}^2$, unit cells of $5 \times 3\,\mu\text{m}^2$, details of around one μm, and a thickness of some hundreds of nanometers, constitute, to our knowledge, the most precise 2D 1/2 auxetics manufactured so far and motivate us to continue researching this subject. The cryo DRIE process can be applied, in a similar way, to the development of several geometries of 2D1/2 memataterials, as well as smart nanostructures and microstructures.

Although we have focused solely on manufacturing issues in our current research, we would like to discuss some important devices and applications that may benefit from the use of micromanufactured auxetic structures because of the outstanding degree of precision attainable by using DRIE. Apart from the application fields detailed in the Introduction of this paper, recent progress in the field of auxetics focuses on their application to the control deformations induced by thermal gradients, especially in plates, shells, spheres, and cylinders (Li, 2013), and to the control of stiffness, especially
in sandwich structures (Grima *et al* 2010). Focusing on the Q37
control of both thermal-induced deformations and structure stiffness is key to enabling additional specific applications of auxetics in several fields of study.

Furthermore, planar auxetic structures have the potential to impact a wide range of applications, from deployable and morphing structures to space-filling composite and medical treatments. The ability to fabricate auxetics using smart

materials can greatly promote their applications, as it may enhance the control of actuation and deployment processes by
Q38 using tunable stiffness responses (Rossiter *et al* 2014). We truly believe that the process detailed here is very adequate for the manufacture of highly precise planar auxetics, and that it can be applied to the manufacture of auxetics using smart materials. In future studies, we hope to address the manufacture of microauxetics using SMAs and polymers. Novel horizons are also opened by the possibility of adjusting the auxetic behavior by means of externally applied electro-
Q39 magnetic fields (Grima *et al* 2013), which should further be explored, hopefully in combination with DRIE manufacturing processes, for the development of magnetomechanical microsystems.

4. Conclusions

In this paper, we have presented a very promising approach for the development of auxetic metamaterials and devices based on the use of DRIE. This process stands out for its precision and potential for use in mass production, and it is widely used in relevant industries linked to electronics and telecommunications. To our knowledge, this research represents the first example where this technology has been applied to the manufacture of auxetic geometries with nanometric details. We have tried to provide interesting details of the design and manufacturing processes, as well as some discussion about our main results, present capabilities, difficulties, and challenges in regards to nanoauxetics. Even though the progressive size reduction of artificially obtained auxetic geometries leads to real mechanical metamaterials and can promote novel applications, other difficulties linked to manipulation and integration into complex devices arise, and further research is needed for taking advantage of nanoauxetic geometries. Future studies will focus on the development of characterization procedures and support devices for addressing the actual auxetic behavior of the geometries obtained. Interesting applications may be based not only on the special stiffness tensors of auxetics, but also on their natural vibration-mode shapes. Using nanomanufactured auxetics, ultra-high resonant actuators may be developed by fixing them to piezoelectric actuators, once present manipulation and integration challenges are tackled.

We foresee relevant applications in several fields, including biomedical and tissue engineering, where the technology may be used to develop active implantable medical devices, minimally invasive surgical actuators, or active scaffolds for dynamic cell culture. This technology may also be used in aerospace and aeronautics, for the development of microactuators and highly accurate deployable structures, or in the fields of telecommunications and optoelectronics, for novel antennae designs, special photonic crystals, and stress-strain electromechanical microsensors. Its use in controlling deformations induced by thermal gradients and for controlling stiffness of materials and structures is also noteworthy, and may promote new applications in the previously mentioned fields of research. We aim to continue our search for new appliances based on these interesting geometries. The process described in this paper can be used for many other families of metamaterials, smart materials, and structures as a way of increasing the precision of available microactuators or microsensors based on the interesting properties of mechanical metamaterials.

Acknowledgments

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Page 1

Q4
Note that the reference citation [Liu 2010] has been changed to [Liu and Hu (2010)] with respect to the reference list provided. Please check.

Page 1

Q5
Note that the reference citation [Griffin 2005] has been changed to [Griffin *et al* (2005)] with respect to the reference list provided. Please check.

Page 2

Q6
Note that the reference citation [Narojczyk 2010] has been changed to [Narojczyk and Wojciechowski (2010)] with respect to the reference list provided. Please check.

Page 2

Q7
Note that the reference citation [Scarpa 2010)] has been changed to [Scarpa *et al* (2010)] with respect to the reference list provided. Please check.

Page 2

Q8
Note that the reference citation [Bianchi 2009] has been changed to [Bianchi *et al* (2010] with respect to the reference list provided. Please check.

Page 2

Q9
Note that the reference citation [Tan 2011] has been changed to [Tan *et al* (2011)] with respect to the reference list provided. Please check.

Page 2

Q10
Note that the reference citation [Gatt 2014] has been changed to [Gatt *et al* (2014)] with respect to the reference list provided. Please check.

Page 2

Q11
Note that the reference citation [Mizzi 2014] has been changed to [Mizzi *et al* (2014)] with respect to the reference list provided. Please check.

Page 2

Q12
Note that the reference citation [Prall 1997] has been changed to [Prall and Lakes 1997] with respect to the reference list provided. Please check.

Page 2

Q13
Note that the reference citation [Grima 2000] has been changed to [Grima and Evans (2000)] with respect to the reference list provided. Please check.

Page 2

Q14
Note that the reference citation [Álvarez Elipe 2012] has been changed to [Álvarez Elipe and Díaz Lantada (2012)] with respect to the reference list provided. Please check.

Page 2

Q15
Note that the reference citation [Xu 1999] has been changed to [Xu *et al* (1999)] with respect to the reference list provided. Please check.

Page 2

Q16
Note that the reference citation [Soman 2012] has been changed to [Soman *et al* (2012)] with respect to the reference list provided. Please check.

Page 2

Q17
We have made a change to this sentence “The special properties of auxetic… traditional micromachining”. Please review our edit.

Page 2

Q18
Note that the reference citation [Alderson 1999] has been changed to [Alderson *et al* (1999)] with respect to the reference list provided. Please check.

Page 2

Q19
Note that the reference citation [Bückmann 2012] has been changed to [Bückmann *et al* (2012)] with respect to the reference list provided. Please check.

Page 2

Q20
Note that the reference citation [Kadic 2012] has been changed to [Kadic *et al* 2012] with respect to the reference list provided. Please check.

Page 2

Q21
Please check the usage of the term [especially those support-less ones] in this context.

Page 2

Q22
Note that the reference citation [Chetcuti 2014] has been changed to [Chetcuti *et al* (2014)] with respect to the reference list provided. Please check.

Page 2

Q23
Note that the reference citation [Pozniak 2013] has been changed to [Pozniak *et al* (2013)] with respect to the reference list provided. Please check.

Page 3

Q24
Note that the reference citation [Lira 2009] has been changed to [Lira *et al* (2009)] with respect to the reference list provided. Please check.

Page 3

Q25
Note that the reference citation [Sun 2013] has been changed to [Sun and Pugno (2013)] with respect to the reference list provided. Please check.

Page 3

Q26
Please check the usage of the term [using the same principle] in this context.

Page 3

Q27
Note that the reference citation [Yang 2011] has been changed to [Yang *et al* (2011)] with respect to the reference list provided. Please check.

Page 3

Q28
Please define the acronym [RF].

Page 3

Q29
We have made a change to this sentence “Finally, a second plasma source… coupled plasma (ICP) substrate.”. Please review our edit.

Page 4

Q30
Please define the acronyms [PMMA, AZ].

Page 4

Q31
Please define the acronym [SOI].

Page 4

Q32
Please define the acronym [HF].

Page 4

Q33
Note that the reference citation [Le Bourhis 2008] has been changed to [Le Bourhis *et al* (2008)] with respect to the reference list provided. Please check.

Page 5

Q34
Note that the reference citation [Wang 2007] has been changed to [Wang *et al* (2007)] with respect to the reference list provided. Please check.

Page 5

Q35
Note that the reference citation [Agero 2010] has been changed to [Agero *et al* (2010)] with respect to the reference list provided. Please check.

Page 5

Q36
Please define the acronym [AFM].

Page 5

Q37
Note that the reference citation [Grima 2010] has been changed to [Grima *et al* (2010)] with respect to the reference list provided. Please check.

Page 6

Q38
Note that the reference citation [Rossiter 2014] has been changed to [Rossiter *et al* (2014)] with respect to the reference list provided. Please check.

Page 6

Q39
Note that the reference citation [Grima 2013] has been changed to [Grima *et al* (2013)] with respect to the reference list provided. Please check.

Page 6

Q40
In SMS 495699 the reference list are numbered whereas the text citations are named i.e. with Name and Year. Please suggest how to proceed further.

Page 6

Q41
Please provide the page range or article number in reference [Agero U, Glazier J A and Hosek M 2010].

Page 6

Q42
Reference [Evans K E] is listed in the reference list but not cited in the text. Please cite in the text, else delete from the list.

Page 6

Q43
Please provide the volume number in reference [Griffin A C, Kumar S and Mc Mullan P J].

Page 7

Q44
Please provide the page range or article number in reference [Le Bourhis E, Morris D J, Oyen M L, Schwaiger R and Staedler T].

Page 7

Q45
Please update the volume and page range in reference [Lim T C].

Page 7

Q46
Reference [Lim T C] is listed in the reference list but not cited in the text. Please cite in the text, else delete from the list.